

NOTE: This document is to be used as a training guide for new chemists in the Massachusetts Department of Public Health's Boston Drug Analysis Laboratory. It is to be used by the Laboratory's senior staff during the initial training period given to all new drug chemists. It is a constantly evolving document that changes to meet the needs of the Laboratory and the requirements imposed on the laboratory by the Commonwealth's courts. It is a DRAFT, and does not cover detailed aspects of all functions of the Laboratory. Any questions or concerns that chemists have about information in this guide should be addressed to the Laboratory Supervisor. This is not a laboratory PROTOCOL.

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Drug Analysis Laboratory Training Guide

The training of new drug laboratory analysts is conducted by senior staff members under the direction of the laboratory supervisor. Training prepares chemists in the analysis of unknown substances submitted to the laboratory. Training will stress the importance and uniqueness of each submitted sample. Since every possible analytical situation cannot be addressed in this guide, it is incumbent upon the analyst to seek guidance from the senior staff when situations arise that are not covered. Following this training, the chemist will be given a written and practical exam to demonstrate proficiency in the testing of unknown substances usually encountered in the laboratory. If the chemist successfully passes both tests, they will be appointed an Assistant Analyst for the Department of Public Health. Each new chemist will receive a copy of the Scientific Working Group for Drugs (SWGDRUG) Code of Professional Practice for Drug Analysts, which can be found in Addendum A

#### Mission of the Drug Analysis Laboratory

The Drug Analysis Laboratory identifies unknown substances for local, state, and federal agencies located in the Commonwealth of Massachusetts. The mission of the Laboratory is “to provide accurate and timely analysis of these unknown substances, and to provide Certificates of Analysis which are prima facie evidence in the courts of the Commonwealth”.

#### ALL NEW CHEMISTS WILL BE INSTRUCTED IN:

- 1) Sample submission process.
- 2) Proper handling of evidence and maintenance of chain of custody.
- 3) Laboratory Safety Procedures.
- 4) Operation of the analytical instrumentation used in the preliminary analysis of drugs.
- 5) Familiarity with Chapter 94-C, of the Massachusetts General Laws.
- 6) Preparation of reagents used in the laboratory.
- 7) State Laboratory Institutes Quality Control/ Quality Assurance (QA/QC) program.
- 8) Recommended Methods of Analysis of the Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG).

## 1) Sample Submission

The Laboratory defines a Case as all items (samples) submitted at the same time for one or co-defendants. Cases are submitted to the laboratory either through the mail or by direct submission to the Laboratory evidence office. Cases can consist of several items\* (samples). All items that are submitted are sealed in a plastic bag and initialed by an agent of the submitting department. The gross weight of the sealed bag is determined by the laboratory evidence officer, and recorded on a laboratory receipt. A Laboratory Control (Evidence) Number is assigned to each item (sample) and is recorded on the drug receipt. A corresponding bar code label is affixed to a manila envelope. The item(s) are placed in the manila envelope. A Laboratory Control Card containing pertinent sample information is generated based on the information on the drug receipt. It is placed in the corresponding manila envelope. The manila envelope is stored in the Evidence Office safe until it is assigned to a chemist for analysis. Entry to the Evidence Office safe is restricted to the Lab Supervisor and designated Evidence Office staff.

\* An item (sample) can be composed of many specimens. Example, a case can consist of 1 bag of powder, 3 bags of vegetable matter and 24 tablets in a vial. This is 1 case, 3 items (samples), and items number two and three each have 3 and 24 specimens (units) respectively.

## 2) Proper Handling of Evidence

All transfers of evidence will be documented either in writing or electronically. The chemists receive samples from a laboratory evidence officer, in the evidence office. Before removing samples from the evidence office, the chemist will perform an evidentiary check. They will check the condition of the evidence bag(s). The chemist will verify that the proper items are contained in the corresponding manila envelope by matching the evidence with the enclosed laboratory control card and with the affixed bar code(s) on the manila envelope. If the evidence bag is intact they will sign for that evidence in the chain of custody logbook, and take possession of the evidence. That evidence will immediately be brought to the individual's work area. The evidence must be stored in a secure manner at all times when in possession of the analyzing chemist.

At the beginning of an analysis, the procedure detailed in Appendix A will be followed, before opening any sample evidence bag. If in the course of analysis the chemist must leave the laboratory area, the evidence will be secured in a locked evidence storage cabinet. Upon completion of analysis, the chemist will seal the evidence in a laboratory evidence bag or pouch. They will clearly mark the evidence pouch (bag) with the laboratory control number and their initials near the middle portion of the evidence pouch. The chemist will also initial their seal of the evidence pouch. The chemist will not initial the intact seals made by the manufacturer. This allows the chemist or a supervisor to determine if the evidence bag has been opened by an outside agency. Evidence should be packaged in a manner that clearly allows the evidence to be visually identified.

When one bag out of several in an item is tested, the tested bag will be identified by a mark such as an X, to indicate which bag was tested. If multiple bags are present for one item number, the bags analyzed will be marked by number. Example. Item number 123456 contains 34 bags. The chemist analyzes six of these bags. The chemist will number the analyzed bags 1 thru 6, and not A thru F. If the item consists of bags in bags, the larger bags will be labeled A B C etc and the bags contained in the larger bags will be labeled A-1,2,3 etc and B-1,2,3 etc.

When testing is completed, a Certificate of Analysis for each item (sample) will be generated by the evidence office. This certificate is based on the results reported on the laboratory control card. Before the chemist returns completed samples to the evidence office, they must verify that all information on the sample evidence pouch, and on the Certificate of Analysis, is correct. The chemist will have the Certificate notarized. The chemist will place the Certificate of Analysis

along with the completed sample into the manila envelope. The chemist will return the completed items to the evidence office with the Laboratory Control Cards. The items should be placed in numerical order.

### 3) Safety

Safety precautions will be followed at all times. Safety glasses and laboratory coats must be worn whenever working at the laboratory bench. Gloves and masks may be used. All needle and syringe samples must be autoclaved before analysis takes place. All guidelines of the State Laboratory Institute Safety Manual will be implemented. Training classes in chemical hygiene and bloodborne pathogens will be attended regularly. The proper use and storage of chemical reagents will be stressed.

### 4) Instruction in Analytical Instrumentation

Chemists will receive instruction in the proper operation of analytical instrumentation used in the preliminary analysis of samples. This training will allow a chemist to perform all preliminary instrumental analyses that the laboratory performs. Training will include, but not be limited to, the use and proper maintenance of Microscopes, Analytical Balances, Gas Chromatographs, and Ultraviolet and Infrared Spectrophotometers.

### 5) Controlled Substance Law

Each chemist will receive a copy of Massachusetts General Law Chapter 94-C (sections 31 and 32). These sections deal with the scheduling of controlled substances in Massachusetts. Each chemist will be trained so that they will be able to identify the class of a controlled substance and will become familiar with the wording of this particular statute.

### 6) Reagent Preparation

All chemists will be instructed in the proper preparation of reagents used in the laboratory. They will also be instructed in the quality control procedures that must be followed to insure the reliability of their reagents. The preparation of all reagents and standards will be recorded in corresponding laboratory notebooks. The chemicals and the identifying lot numbers should be noted. The information will be initialed and dated. Reagents will be tested with known substances to verify reliability.

### 7) QA/QC Procedures

Each new chemist will become familiar with the overall Quality Assurance and Quality Control (QA/QC) procedures of the laboratory. The goal of the QA/QC program is to ensure that established laboratory procedures are followed at all times. Random QC checks of several of the laboratories completed samples will be performed on a monthly basis to insure adherence to standard procedure. This will involve examination of paperwork and / or reexamination of selected completed items. Proper storage of analytical data in chronological order will be emphasized. A supervisor must be able to retrieve all data in the absence of the chemist. All notations made by the chemist should be legible. Any errors should be lined out and initialed, by the chemist. When data is to be sent out of the laboratory to interested parties (Assistant District Attorney's, Court Officials etc.,) it will be reviewed by the laboratory supervisor or a senior level chemist before being sent. Periodically, a formal Quality Control discussion will take the place of the monthly audit.

### 8) Methods of Analysis

The Drug Analysis Laboratory follows the Recommendations of the Scientific Working group for the Analysis of Seized Drugs (SWGDRUG) for its methods of analysis. Addendum B

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## Categories of Samples

- A. Powders and Substances
- B. Pharmaceuticals
- C. Vegetable Matter as Suspected Marijuana
- D. Residues
- E. Needles and Syringes
- F. Others (includes)
  - 1) Alcohols
  - 2) Nitrites
  - 3) LSD (lysergic acid diethylamide)
  - 4) Tetrahydrocannabinol (THC) Hashish, Brown Substance
  - 5) Steroids
  - 6) MDMA (methylenedioxymethylamphetamine), MDA and other Phenethylamines
  - 7) Phencyclidine (PCP) submitted as vegetable matter
  - 8) Mushrooms (psilocybin)
  - 9) Ketamine liquid
  - 10) Gamma-Hydroxy Butyric acid (GHB), Gamma-Butyrolactone (GBL)
  - 11) Inhalants (nitrous oxide)
  - 12) Opium
  - 13) Bio-terrorism samples
- G. Negative Samples
- H. Resubmitted samples
- I. Not Tested Samples
- J. Found Drugs

## Analysis of Samples

### Powder and Substance samples

Cocaine HCl, Cocaine base (Crack), Heroin, Amphetamine and Methamphetamine, etc.

1) Verification of Assigned Item (Sample)

A) See appendix (A)

2) Gross Examination and Description of Item

The following information will be recorded on a Sample Analysis (Powder) Reporting Sheet and will include:

- A) Sample Number and Submitting Agency
- B) Verification of evidence office gross weight
- C) Number and Container type in sample
- D) Powder or Substance
- E) Gross weight of exhibit (all specimens will be weighed)
- F) Visual determination of similarity of specimens by gross examination(size, color, container type)
- G) Determination of net weight
- H) Test results and notations
- I) Preliminary result finding and date
- J) Confirmatory result, GC/MS operator, and date

3) Sampling Procedures

Testing of a representative number of specimens in each item (sample) submitted should be done according to the sampling plan (appendix B).

4) Net weight

Net weights will be determined on all powder samples that are amenable to being weighed (excludes residues). For each suspected trafficking case, the balance the chemist will use is to be checked with known weights to demonstrate its accuracy. This demonstration of the balances accuracy will be noted on the analyst's powder reporting sheet. A minimum of 2 standard weights will be used. The serial number of the balance used will also be recorded on the analysts reporting sheet.

Net weights can be determined by either of two methods

- A) Direct weighing of the substance: The substance is emptied from its container into a properly tared weigh boat or onto a piece of tared weighing paper. The net weight is recorded on the chemists analysis sheet
- B) By Difference.... The gross weight (substance and container) is determined and recorded. The substance is emptied out and the weight of the empty container is recorded. The weight of the empty container is then subtracted from the gross weight to determine the net weight. In the case where the sample may be a plastic bag inside another plastic bag, it should be separated and only the actual bag containing the substance should be weighed for the gross weight. The weight of the empty container is then subtracted from the gross weight, to give the net weight. All calculations, and the net weight will be recorded on the chemist's analysis sheet

- C) The net weight of a multiple specimen sample is calculated by following the plan in appendix C. The chemist will record all weighings on their analysis sheet.
- D) The net weight is also reported on the Laboratory Control Card.
- E) The number of units tested from the population of a sample will be recorded on the Laboratory control card along with the preliminary result.

## 5) Preliminary Tests

Preliminary tests help the chemist in the presumptive identification of unknown samples. They will be performed on all powder/substance samples, and can include the following: Color tests, Microcrystal tests, or Analytical Instrument tests.

A) Color ( Spot ) Tests: Color tests will be performed on items when sufficient material is available. Color tests, are usually performed by placing a small amount of the sample into a depression on a porcelain (spot) plate and adding a particular reagent (appendix I). Results can be reported as Positive (+), or Negative (-), or the various resulting color changes can be described.

Marquis, Froehdes, Meckes & Cobalt Thiocyanate (Cob/Thio) color tests will be done on all powder samples.

- a) A negative color test is one that results in no color change.
- b) If the Cobalt Thiocyanate test result is negative, an acidified Cob/Thio test will be performed. This is done by adding a drop of dilute hydrochloric or acetic acid to the solution in the Cob/Thio depression of the spot plate.

Examples of typical color test reactions are:

### Heroin

Marquis.....Purple or Positive (+)  
Froehdes.....Purple to Slate or Positive (+)  
Meckes.....Green or Positive (+)

### Cocaine

Cobalt Thiocyanate...Flaky Blue or Positive (+)

### Cocaine base (Crack)

Cobalt/Thiocyanate... Weak blue or Negative (-)  
Acidified Cob/Thio.... Stronger blue or Positive (+)

### Amphetamine or Methamphetamine

Marquis test.....Strong Orange to Brown (this result will be spelled out). Do not put down (+) for a result, as this will be confusing with a (+) purple result for Opiates. If the Marquis test results in a orange to brown reaction, then the chemist should proceed to UV or GC testing for presumptive identification of an Amphetamine type drug. See Methods Addendum.

NOTE: For a suspected cocaine item with multiple units, and all the units are similar, all screening color tests will be performed on at least one specimen. If the Marquis, Froedhes and Meckes tests are negative in the initial testing, and the Cobalt test is positive, then only the Cobalt test need be done on the majority of the other tested specimens.

See Clarke's Isolation and Identification of Drugs, Volumes 1,2 and 3, and the DEA Forensic Chemists Training Manual for details on procedures and typical positive color reactions of other drugs.

#### B) Microcrystal Tests

Microcrystal tests involve placing a small amount of sample on a glass microscope slide and adding specific crystallizing reagents. The resulting precipitate is then examined under a polarizing light microscope (4X or 10X magnification), and characteristic crystals are observed. Microcrystal tests will be performed on ALL presumptive Cocaine samples (those giving a positive Cobalt test), or any substance that gives a similar retention time to a Cocaine standard by Gas Chromatography analysis.

- 1) Gold Chloride crystal test
- 2) TLTA (Di-p-toluoyl-d-tartaric acid monohydrate) crystal test

(See Appendix H)

#### C) Instrumental Tests

- 1) Gas Chromatography (GC) Appendix G
- 2) Ultraviolet Spectroscopy (UV) Appendix U

Samples resulting in positive color test reactions that are not typical to those of Cocaine or Heroin, will be run by a screening instrumental technique, (either UV or GC), to determine if other controlled substances may be present.

Example. A sample which when tested yields an orange to brown Marquis color test, or a sample giving a blue result with cobalt thiocyanate, but giving no positive crystals for cocaine, will be run as specified in appendix G-1 or U-1, using the appropriate GC or UV testing conditions.

#### 6) Confirmatory testing.

Preparation for GC/MS confirmatory analysis

#### COCAINE and HEROIN

- a) A sample that tests presumptively positive for Cocaine or Heroin by screening tests, will be run by Gas Chromatography-Mass Spectroscopy (GC-MS) for confirmation. A small amount of the sample (3-5 mg) will be placed in a 1.8-2.0 mL GC vial, and it will be

dissolved and brought to volume in a solvent such as methanol, ethanol, methylene chloride etc.). The vial will be crimp sealed, and numbered.

- b) The chemist doing the preliminary testing (primary chemist) will write their preliminary findings on the Laboratory Control Card.
- c) A GC-MS chain of custody control sheet will be prepared (appendix E). The control sheet and the control card for each sample, along with the sample GC vials, will be submitted to the GC-MS lab for testing. A chemist in the GC-MS laboratory will indicate on the control sheet when they received the vials for testing.
- d) Usually, all sampled units that test positive by preliminary methods, will be confirmed by GC-MS. However, the laboratory supervisor or a senior analyst may adjust the number that will be sent for confirmatory testing. They will initial the analysis sheet of the chemist and note that they have made an adjustment to the routine procedure. Example 20 specimens out of a total of 50 are weighed to determine an accurate estimated net weight. The supervisor may determine that only the square root (8), or 10% (5), need be submitted for GC-MS confirmation.
- e) Suspected Cocaine and Heroin sample GC vials will be segregated from other sample vials

#### Other SUBSTANCES

- a) If preliminary tests indicate the possible presence of a controlled substance, other than Cocaine or Heroin, then a GC-MS confirmation test will be run. Depending on the suspected drug, a chemical cleanup may be necessary before submitting to GC-MS lab for confirmation. See Clarke's Isolation and Identification of Drugs, the Merck Index, or addendum M, to determine the solubility characteristics of the suspect drug. A sample will be prepared for GC/MS. The primary chemist will indicate their suspected findings as in (b) above.
- b) A control sheet will be prepared as in Appendix E and the sample vials will be submitted to GC-MS.

#### 7) Confirmatory Testing (GC/MS):

The sample aliquots (GC vials), will be tested by a second chemist using GC-MS for confirmation. After this analysis, the GC-MS operator will record their results and the date completed on the GC-MS control sheet. The GC-MS chemist will return the sheet to the primary chemist. The primary chemist will note the result, the GC-MS operator, and the date completed on their analysis sheet.

The GC-MS operator will also write the results and the date analyzed on the Laboratory Control Card. They will initial the card, and if the confirmatory test matches that of the preliminary testing, they will send the control card to the evidence office.

If the GC-MS result is different from the preliminary result, the submitted vial, the control sheet, and the control card will be returned to the primary chemist for re-examination of the sample.

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- 8) For a powder/substance sample to be reported as containing a controlled substance, both the preliminary and the confirmatory tests must agree. For Cocaine, the preliminary test must include positive results for the Gold Chloride and the TLTA microcrystal test.

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9) A Certificate of Analysis will be generated by the Evidence Office staff for each item based on the information on the control card. Both the primary chemist and the GC-MS operator will verify the results on the certificate. If correct, both chemists will sign the certificate. The certificate will then be notarized.

10) ANALYSIS (powder) SHEETS will be kept by the primary chemist and stored in chronological order. A supervisory chemist must be able to retrieve all data in the case of a chemist being absent.

( Paper copies of all preliminary instrumental data that is generated in the analysis of a sample will be saved by the primary chemist )

11) Handling of Negative Powder Samples. (see section G)

12) Determination of Cocaine Base (crack), from Cocaine HCl (salt form)

If the salt form of a Cocaine sample needs to be determined, the following tests will be performed.

- 1 Cobalt Thiocyanate color test
- 2 Microcrystalline test
- 3 Gas chromatography analysis
- 4 Infra Red analysis

A) CobaltThiocyanate/Acidified Cobalt Thiocyanate color test-If the substance is Cocaine Base (crack), a weak or negative result should occur with the Cobalt Thiocyanate reagent. A pronounced blue color result should occur with the addition of weak Hydrochloric acid. This is due to the different solubility of cocaine base from the salt. If a typical blue color results from the Cobalt Thiocyanate reagent alone, then the sample could be Cocaine HCL, a mixture of Cocaine Base and Cocaine HCl, or either form of cocaine with an interfering substance such a Procaine-HCl.

B) Microcrystal test will be performed. See (appendix H)

C) GC analysis will be performed to determine if other compounds are present in the sample\*. If only a cocaine peak is detected by GC analysis, then an Infra-Red (IR) analysis will be performed as described in

section (D) below. If a significant GC peak (1/10 the area of the cocaine peak or greater) is found, the sample should be handled as follows.

\* If a GC/MS analysis has been performed on the specimen to be tested, then the chemist may review the original GC/MS to see if interfering substances are present in the specimen. If interfering substances were found in the GC analysis, the sample will be sent to GC-MS to determine the interfering substance. A chemical cleanup, such as a cyclohexane extraction, will be used to remove the interfering compound. This cleanup must not change the salt form of the suspected cocaine. The senior staff should be consulted on this type of sample.

D) The Infra- Red (IR) analysis should be performed in such a manner, that no change in the chemical form of the sample takes place. The sample can be mixed with KBr and a pellet made and IR analysis done. Also, the sample may be analyzed by diffuse reflectance (no pellet) IR. The prepared sample will be scanned from a wavelength of approximately 4000-600 nm, and the resulting spectra compared to that of a known Cocaine base standard. The spectra of the sample should match the known standard spectra in general appearance, and at least 4 major peaks should be consistent with the standard spectra (+ or - 4) wavelengths. The chemist will use a library search if available with the instrument, and will also visually compare the sample spectra with that of a previously run standard.

## B. Pharmaceutical Analysis

### 1) Verification of Assigned Sample (appendix A)

### 2) Gross examination of sample

A) The chemist will count the tablets (including partial tablets) and this will be recorded by the chemist.

If there are several hundred units in the exhibit, the chemist may estimate the count in the following manner

- a) Determine the net weight of entire exhibit
- b) Determine the average net weight of three (3) tablets
- c) Divide the total net weight of the tablets by the average weight of one.

All computations will be recorded on the analyst's reporting sheet.

B) A net weight will be determined on all pharmaceutical items, and the result will be recorded on the chemist's reporting sheet.

C) The markings and a description of the tablets will be recorded on the chemist's sheet.

Information will be recorded on a Powder Sheet or on a Pharmaceutical Reporting Form. These forms will be kept in chronological order, either by date completed or date assigned. Any data generated in the analysis of an item, including GC/MS control sheets, will be kept by the primary chemist.

### 3) Ballistic Identification\*: All capsules and tablets will be examined ballistically for manufacturer's markings, shape, color, size etc. The various identification manuals listed in appendix F should be used to compare and identify the sample.

\* The Laboratory uses the term Ballistics to indicate testing that involves matching the markings, colors, and shapes of tablets to known pharmaceuticals.

\*\*The results of computer searches will be printed out and saved with the sample analysis sheet\*\*

For commonly occurring tablets such as Oxycontin 80 mg and Suboxone 8mg (Buprenorphine), a preprinted copy of the Micromedex search result may be used. The current date of analysis will be recorded on the preprinted search

4) If the tablets are determined by ballistics to be a class E, or a non controlled substance, proceed to number 9 of this section.

5) If the tablets can not be identified by ballistics, then they will be analyzed chemically as an unknown powder sample. One to Three tablets should be tested.

6) If there is more than one tablet, and they are determined by ballistics to contain OXYCODONE, HYDROMORPHONE, BUPRENORPHINE, MORPHINE, or CODEINE (class B), then the net weight will be reported on the Laboratory Control Card, as well as on the chemist's reporting sheet. All multiple tablet OXYCODONE, HYDROMORPHONE, BUPRENORPHINE, MORPHINE, and CODEINE (class B) pharmaceutical items will have a net weight reported on the Certificate of Analysis. A single tablet that is an opium derivative, or any

other pharmaceutical items do not need to have the net weight reported on the Certificate unless this is requested by the District Attorney's office.

The balanced used by the chemist for items that are greater than 14 grams will be checked by the analyst. At least two known weights will be used to verify the balance operation, and the balanced used will be recorded on the analyst's work sheet.

7) If tablets are identified by ballistics as containing a class A, B, C, or D substance, then a chemical analysis by GC/MS will be performed for confirmation.

A chemical cleanup may be required for some pharmaceuticals before submitting to the GC/MS lab. Typical cleanups can be found in several references, including Clarke's Isolation and Identification of Drugs, The Merck Index, and in addendum (M).

Based upon the concentration of controlled substance in a tablet, an aliquot of sample will be prepared in a GC vial to contain approximately a concentration of 1.0 mg/ml, of the suspected drug of interest. The primary chemist will write the presumptive identification of the controlled substance on the laboratory control card, and will submit the aliquot of sample to the GC/MS laboratory as in appendix E.

If the results of the GC/MS analysis confirm the presence of the suspected controlled substance, then the GC/MS operator will return the card and the GC/MS results back to the primary chemist. The primary chemist will record and check results and send the card to the evidence office for a Certificate of Analysis.

If the GC/MS results are not suitable, for example, the result doesn't match the preliminary result, or the concentration of the submitted GC vial does not allow for a positive MS result, then the GC operator will return the vial and the card back to the primary chemist so that the primary chemist can adjust the concentration of the vial or recheck initial work.

8) Pharmaceuticals that are chemically analyzed will follow the sampling plan listed in Appendix B, section 3.

Note- Some derivatives of Opium are listed in the Massachusetts law as being class B, C or E. This is determined by the concentration of the drug. Example: Hydrocodone and Codeine. See Addendum L, MGL chapter 94 C.

When a chemist is not clear on the class of any pharmaceutical analyzed, they will check with a senior analyst, or the laboratory supervisor.

-If a chemist uses more than one tablet for an analysis, they will note the number used on their reporting form.

9) If a sample is identified by ballistics, and is determined to be a manufactured pharmaceutical containing a class E substance according to Chapter 94 C, of the MGL, or a substance that is not controlled, then the sample can be identified solely by this method (ballistics).

a) For items that are identified solely by ballistics, the certificate of analysis will state that the specimens (capsules, tablets etc.) contained (X-chemical name-), and was identified by Appearance and Labeling, (A & L).

For samples identified solely by Appearance and Labeling, the chemist will follow the following procedure.

The chemist will perform a computer search such as the Micromedex Search. If the tablet(s) can be identified in this manner, the chemist will print out the computer search result and keep the hard copy for their records. If the computer search lists multiple drugs with similar markings, the chemist will check the differences in color, shape, markings, and size, before determining the drug present. If in doubt, the chemist will check with a supervisor. If a computer search is not available, the chemist may use a published reference guide (listed in appendix F, to discern the tablets identity. If successful, the chemist will note the reference guide used. When a reference guide other than a computer search is used, the chemist will notify another analyst as to their finding. The second chemist will verify the findings in the reference guide, and will initial the primary chemist's findings on the primary chemists reporting form.

\*\*\*Any identification done solely by appearance and labeling, that does NOT have a computer printout, will have two chemists' initials as to the identity clearly marked on the reporting sheet. \*\*\*

If a chemist encounters a marked tablet that cannot be identified in any of the search references, they should analyze it as an unknown. If any positive results are confirmed by GC/MS, the chemist should record this tablet, its markings, and the result in the State Lab Pharmaceutical Logo binder for future reference, as well as report their findings to the evidence office.

### C. Marijuana Vegetable Matter Analysis

- 1) Verification of assigned sample (Appendix A)
- 2) Gross examination of sample including exhibit gross weight
- 3) Information (gross weight, date, tests performed, and results) will be recorded and initialed on the Control Card, on a reporting sheet, or in bound book. If the control card is used, a Xerox copy of the card will be made.
- 4) A net weight will not be done on routine Marijuana samples
- 5) Sampling plan (Appendix B-3)
- 6) Tests:
  - a) Macroscopic identification of vegetable material and Microscopic identification of cystolith and glandular hairs.
  - b) Chemical test (Modified Duquenois- Levine). See procedure in the Official Methods of Analysis of the Association of Official Analytical Chemists, Thirteenth Edition

If the microscopic, macroscopic, and Duquenois-Levine tests are positive, the sample can be reported as Marijuana.

If all tests are negative, and the sample does not have a pungent odor, then the sample may be reported as negative. If the sample has a pungent odor, it should be tested for PCP.

For marijuana items that have net weights of approximately one pound (454 grams) or more, the chemist should check with a supervisor to see if more testing is necessary (GC or GC/ MS analysis).

If the result of the Macroscopic and Microscopic analysis are inconclusive, but the Duquenois test is positive, then the sample will be further tested as follows:

- A) Place 10-20 milligrams of vegetable matter into a 16x100ml glass tube
- B) Add 2-3 ml of solvent (eg. Methanol, Ethanol, or Petroleum Ether) to the tube and cover
- C) Let sample sit in solvent for approximately 20-30 minutes with occasional mixing
- D) Remove 2ml into a standard GC vial, crimp seal and number vial
- E) Run aliquot by appropriate GC method with delta-9-THC standard (appendix G).
- F) If the GC analysis results in a positive retention time match to delta 9 THC, then the vial will be submitted to GC-MS for confirmation (appendix E).
- G) If GC testing indicates a substance other than THC, then a Cocaine standard should be run, to determine the relative retention time of the substance. The original vial will be sent to GC-MS for confirmation (appendix E).
- H) If no GC peaks are found, the sample will be reported as NEGATIVE.

#### D. Residues

Residue samples may contain such small amounts that the analyst is not able to perform complete screening tests. The analyst may then follow an abbreviated screening plan (example, no performing of color test). The chemist should perform a preliminary GC analysis, and then proceed to confirmatory testing.

If a residue item is part of a case that contains an item (sample) that has tested positive, then the residue will be handled as a NOT TESTED sample (section I).

\* It is imperative that glassware, work area and utensils, be clean before performing a Residue analysis\*

##### A) Residues-- excluding suspected Marijuana residues (marijuana pipes) or Needles and Syringes

Examples: cooker (bottle) caps, spoons, straws, cocaine pipes

- 1) Verification of assigned sample (appendix A).
- 2) Examine various objects in the sample. Note and describe the sample. Example (Bottle caps, straws, tubes, spoon, bags etc). Choose the object that is most conducive to being analyzed (for example a spoon with heavy residue). If the sample consists of residue in many bags, the chemist should choose several and perform one or two preliminary tests on those. If preliminary tests are positive the chemist may composite the bags as one sample. Analyze this specimen as a powder sample. Net weight not necessary. If sufficient sample is present, scrape off enough for testing, and proceed as with powder testing (net weight not necessary). If a positive result is attained, do not analyze any of the other specimens. If the tests are negative, choose another specimen in the sample, which is most conducive to analyzing. If the sample is still negative and other specimens remain, then a senior level chemist should be notified, to determine what further testing should be done.
- 3) Some residues will require a solvent rinse in order to retrieve a sufficient amount for testing. A minimal volume is used (approx. 0.5 ml- 1.0 ml of Methanol). Rinse into a clean test tube (12 x 75 mm). Use half the solvent rinse for preliminary GC testing (see appendix G-3).

\*\*The methanol used for the GC analysis of a suspected crack pipe will be acidified with 2.8n Hcl or 20% acetic acid\*\*

- 4) If the GC result is positive by retention time for Cocaine, place two separate drops from the remaining rinse onto two sections of a microscope slide and allow to dry. A Gold-CL test will be run on one drop, while a TLTA test will be performed on the other. If both crystal tests are positive for cocaine, the original vial that tested positive by GC analysis will be sent to the GC-MS lab for confirmation. If crystal tests are inconclusive or negative, a senior level chemist should be notified or the tests should be rerun.
- 5) If the preliminary GC testing indicates a substance other than cocaine then the sample will be sent to GC/MS for confirmation. The chemist will note on the GC-MS control sheet the relative retention time of the sample as compared to a cocaine/codeine standard.

##### B) Residue Pipes... Suspected ( Marijuana)

- 1) Verification of sample ( appendix A)
- 2) If vegetable matter is present, but unidentifiable macroscopically as marijuana, using the smallest amount possible, scrape a small portion into a casserole dish and also into a clean test tube. Perform a microscopic exam and a Duquenois test on the portion in the casserole.

To the portion in the tube add a small amount of methanol or ethanol (approx 0.5 ml) and run a screening GC test along with standard delta 9 THC. If the Microscopic and Duquenois tests are positive, and the GC analysis matches the THC standard, the sample can be called Marijuana. If the Duquenois or the microscopic test is inconclusive, then the sample must be submitted to GC-MS for testing before it can be called positive.

- 3) If residue exists, but no identifiable vegetable matter is present for microscopic testing, rinse some of the residue into a clean test tube with approximately 0.5-1.0 ml of solvent and run screening GC test as above.
- 4) If sample retention time is positive to THC standard by GC, then the vial will be submitted to GC/MS for confirmation testing in the prescribed manner (appendix E). It is not necessary to get a positive Duquenois result if a GC-MS test is performed and is positive.
- 5) If negative by GC testing, the sample is reported as negative.

#### C) Not Tested Residues

##### \*\* ATTENTION\*\*

If a residue, or a group of residues is submitted with a specimen that has tested positive, the residue will be treated as a NOT TESTED SAMPLE (section I).

In some circumstances, even when a Positive result has been attained in the case, a residue may need to be tested for probable cause or some other reason. If a special request for testing is made, the residue will be tested. The chemist should check with the Evidence Office staff or a senior level chemist before doing this analysis.

#### E. Needles and Syringes \*\*\*\*

- 1) Needles and Syringes will be stored in rigid capped tubes beginning at the time of submission to the Lab.
- 2) N & S will be returned to police agencies as " NOT TESTED" \*\*\*\*\*specimens unless a special request for analysis is received. A special request for testing will be honored if the N&S is the only item in the case, or if a compelling reason for analysis is made by an Assistant D.A, or the submitting agency.
- 3) If analysis is to take place then refer to N&S handling attachment(Addendum M)
- 4) After autoclaving, the N&S should be rinsed with solvent and tested by GC. If positive results occur by GC testing the vial will be submitted to GC-MS. If possible, microcrystal tests will be performed on the sample if GC-MS results indicate the presence of Cocaine.

\*\*\*\*\* AS OF 1/01/07 NEEDLES AND SYRINGES WILL NOT BE ANALYZED\*\*\*\*\*

#### F. Others

All other types of samples will be examined by preliminary tests and if positive, they will be confirmed by GC-MS. The chemist will follow the general procedure as described in appendix A. Sampling will be determined at time of analysis and a senior level chemist will be involved in determining how many specimens will be tested. Appropriate testing procedures will be found in Addendum M.

## G Negative samples

### 1) Powders

The following should be negative before a powder sample is determined to be NEGATIVE

#### a) Color tests

- 1) Marquis
- 2) Froedhes
- 3) Meckes
- 4) Cobalt Thiocyanate and Cobalt Thiocyanate w/acid
- 5) Dilly-Koppanyi test for Barbiturates

The Dilly-Koppanyi test is done by placing a small amount of sample, (2-5 mg) into a glass test tube (approx 16 x 100mm). 1-2 ml of 5% isopropylamine and 1-2 ml of Dilly Koppanyi reagent are then added to the tube. A strong purple color will quickly develop if any of the barbituric acid derivatives are present.

#### b) Visual (Organoleptic)

A visual examination of the specimen should be done. If the sample has the appearance of a crushed tablet or a consistency that may indicate an illicit substance, further testing by GC or UV should be done.

If any of the above tests are questionable or give some type of positive reaction, then further testing by an instrumental technique such as UV or a GC should be done on the sample.

If the color, Dilly, and organoleptic tests are negative, then the sample can be reported as negative for controlled substances.

For GC testing, (see appendix G).

For UV testing (appendix U)

If results for GC or UV testing are positive, the substance should be further tested by GC/MS.

If results of GC or UV are negative, then the sample can be reported as negative.

#### c) Sampling Plan for Negative Powders see (appendix B)

### 2) Vegetable Matter

If a vegetable matter sample is negative by all preliminary tests (Microscopic, Macroscopic, and Duquenois), and it has no suspicious pungent odor (PCP) it can be called negative. If the chemist suspects the possibility of a controlled substance, they should proceed to GC testing as in appendix G-2. The sampling plan under appendix B for powders will be followed, except that composites may be done for UV and GC testing.

### 3) Tablets (unmarked pharmaceuticals)

If tablets/ capsules are similar in appearance, then at least 3 will undergo color testing to see if they are similar. If color tests are the same, then only one specimen will be further tested. A portion of the sample will be dissolved in methanol or ethanol and will be tested by GC (see appendix G). A Dilly-Koppanyi test for barbiturates will also be performed. If the GC analysis and the Dilly test are negative, then the sample can be called negative for controlled substances.

A Negative result means that no controlled substance was identified

#### H) Resubmitted Samples

The evidence office will notify the supervisor of any samples resubmitted to the Laboratory. If the supervisor is not available, then a senior level chemist must be notified. If a fax, or a letter requesting reanalysis exists, it will be saved by the chemist for chain of custody purposes. A copy will be given to the laboratory supervisor. The chemist that performed the original analysis will check the resubmitted sample and verify that it is in the original condition as when it originally was signed out of the laboratory. They will note this on their analysis sheet. The analyst and the laboratory supervisor (or a senior analyst) will determine what additional testing will be done. If the original chemist is unavailable, the Laboratory supervisor and another chemist will open and analyze the sample.

#### I) Not Tested Samples

If a sample is not tested, the original sample submission bag should not be opened. The evidence office gross weight will be checked as usual. The unopened item will be placed in one of the laboratory's evidence bags and the chemist will mark the bag with the sample number, their initials, write NOT TESTED on the bag, seal the bag, and initial the seal. A record of the not tested sample will be kept either as a separate sample, or it can be recorded on a companion sample sheet, for that case.

Reasons why some items will not be tested may include:

- 1) Several similar items submitted in the same case, that will not add to the preponderance of evidence
- 2) Police need items NOT TESTED for Destruction purposes
- 3) Resubmittals for Defense challenges

#### J) Found Drugs

In some instances samples will be submitted to the laboratory after being found, and no defendant is involved. The Police will submit the samples as found drugs. The chemist will handle the sample as a NOT TESTED sample.

#### K) Daily Analysis Housekeeping:

- i. Accuracy of Balances will be demonstrated with standard weights before the initial weighing of the day. At least two different weights will be used.
- ii. Workbenches will be wiped clean before each analysis
- iii. Workbenches should be sprayed with disinfectant at the end of the day
- iv. Sampling utensils will be cleaned between samples
- v. Reagents and sampling equipment will be stored in clean closed areas.
- vi. Negative control will be run on color test reagents before analyzing first powder sample of the day.
- vii. Spot plates will be rinsed immediately after use into a hazardous waste container or they may be placed into a bucket containing detergent and warm water. All water will be collected as hazardous waste.
- viii. Marijuana waste will be neutralized immediately after testing is completed.
- ix. Negative control will be run with Marijuana testing reagents before the first analysis of each day.

## Appendix A- Verification of Sample

Chemists will receive items that need to be tested in the evidence office.

An evidence officer will scan the bar code of the envelopes containing drug items (sample) which are to be assigned to the chemist.

A list of the items assigned to the chemist will be generated.

The chemist will check the evidence before they leave the evidence office area.

The chemist will check that the envelope, the drug control card, and the packaged evidence, correspond to each other. Obvious discrepancies between the card, the envelope and the evidence, should be corrected with the evidence office personnel at this time.

If information is correct, the chemist will sign the chain of custody log for the corresponding sample, and take custody of the evidence.

Before chemists open evidence and begin analysis they will:

- 1) Verify that the samples in the manila envelope correspond to the samples listed on the envelope, and to the control cards.
- 2) Look for obvious errors, such as typographical errors. (Example, Control card says 4 plastic bags, when there are 24 bags present, or the submitting town on card is different from that on envelope, etc.).  
If errors are found DO NOT OPEN BAG: NOTIFY SENIOR STAFF

- 3) Verify the Evidence Office gross weight. Chemists should use a guideline of plus or minus 5 % difference from the weight on the control card.

If greater than 5% difference, DO NOT OPEN EVIDENCE BAG-NOTIFY SENIOR STAFF

- 4) Verify that each item bag is sealed and has the initials of the submitting police officer on the seal.
  - a) If the seal is intact, but is not initialed, the chemists will make note of that error in their records. The evidence office weight must be correct. They may continue the pre-analysis process.
  - b) If the bag is not sealed, the chemists will not proceed to analysis. They will notify the Lab Supervisor or a senior chemist of the improper condition of the sample submission bag. When possible, a laboratory evidence officer will notify the submitting department of the problem.

\*If an error is discovered in any of the above pre-analytical checks, or if some other abnormality in the sample is found, then THE CHEMIST SHOULD NOT OPEN THE SAMPLE BAG. The laboratory evidence officer a senior level chemist should be notified.

- 5) If all of the above conditions are satisfactory, then the chemist will open the sample bag in a manner that maintains the integrity of the sealed and initialed end.
- 6) If a chemist opens the sample bag and discovers a discrepancy, such as the unit count is incorrect, or the control card indicates a bag instead of a packet, he/she should notify a senior level chemist. If the discrepancy requires a change to the card, then the senior chemist and the primary chemist should check the receipt. The senior level chemist will note that the receipt has been checked and that a corrective action has been taken and they may make a correction to the card. The supervisor will make a note on the back of the laboratory control card, and initial and date the back of the card. The correction will be then be made to the front of the card.

- 7) If the chemist opens the evidence bag and discovers that the item consists of specimens that are not similar, he/she may handle the item as an A and B. For example, item 654321 contains two specimens, one is powder in a plastic bag, and the other is vegetable matter in a glassine bag. The chemist should handle the item as 654321 A and B. A senior level chemist should be notified before a card is changed to an A and B item.
- 8) If the chemist finds different types of containers submitted as one number, they should check with the supervisor. If both containers contain the same substance, the sample can be handled under one certificate. The description of the containers can be recorded as "containers".

## Appendix B- Sampling Plans

The Laboratory uses sampling plans that are statistically based, such as the Hypergeometric method, and those that are non statistically based, such as the testing of one, testing of 10%, or the testing of the square root of the total population.

Note: When several items of similar class are involved in one case, the laboratory may reduce its sampling plan, NOT TEST items, or in the case of pharmaceuticals, identify items by appearance and labeling

### 1. Powders / Substances.

Units/Specimens are "similar" by size, shape, and appearance of contents.

1) Non-Trafficking Items: - chemist estimates the net weight to be less than 10 grams

a) 1 to 10 units (inclusive)... 1 will be analyzed

b) 11 to n units..... 10 % of the specimens may be tested, up to a maximum of 20.

The chemist may sample more with the approval of the Lab Supervisor or a senior level chemist

2) Trafficking items- chemist estimates that the net weight will approach 14 grams or greater

#### a) NON-STATISTICAL SAMPLING PLAN

a) Chemist will do complete analysis on at least the square root of the total number of units submitted, up to a maximum of 30. Example- Powder in 59 bags with a gross weight of 16.20 grams, then chemist will do at least the square root (8).

b) A trafficking case may involve separate item numbers from the same case. Example, three item cards- 123456- powder in 7 bags, gross weight 8.24 grams.  
123457- powder in 10 bags gross wt. 7.60 grams, and 123458- powder in 2 bags, gross wt 6.42 grams.

In this case a minimum of the square root of each sample number should be tested -- # 123456 (3 bags), -- # 123457 (4 bags), and-- # 123458 (one bag), since the total estimated net weight, may approach 14.00 grams.

#### b) STATISTICAL SAMPLING PLAN

\*\* For trafficking samples where a statistical inference will be made as to the condition of the entire sample, then the HYPERGEOMETRIC statistical sampling method will be used. The sampling schedule is found on page 25.

Units/Specimens not similar by size

- Non Trafficking: if the sample contains specimens of varying sizes (example, 5 small, 4 medium, and 10 large bags) each type should be separated and a gross weight of each size unit should be done. At least one of each size will be analyzed, and a calculated net weight will be done. (see Appendix C)

-Trafficking: If the units/specimens are of different size, then a representative sampling of each size unit will be done and a net weight calculated from the samples units.

\* In any instance when a testing chemist is in doubt as to the number of units/specimens to test, then a senior level chemist or the Lab Supervisor should be contacted\*

## HYPERGEOMETRIC SAMPLING PLAN

## 2) Vegetable Matter (Marijuana)

The Massachusetts Controlled Substance Law, Chapter 94, was changed in regards to Possession of Marijuana on January 2, 2009.

### 1) Single Item (Sample Number)

#### a) Single bag:

If the evidence office gross weight is less than 28 grams, then no net weight will be determined.

If the evidence office gross weight is greater than 28 grams, and the chemists' gross weight indicates that the net weight may be greater than 28 grams, then the chemist will proceed to determine a net weight of the vegetable material. If the net weight is greater than 28 grams, it will be reported on the certificate.

#### b) Multiple bags:

If the item has an evidence office gross weight less than 28 grams, then no net weight will be determined. 10 % of the bags will be analyzed.

If the evidence office gross weight is greater than 28 grams, and the chemist believes that the total net weight of all the bags is greater than 28 grams, then the chemist will test and determine the net weight of enough bags to insure that a net weight of material greater than 28 grams has been tested. The net weight of the bags tested will be reported on the certificate. At least 10 % of the bags will be tested.

### 2) Multiple Items (different numbers/ same case)

If the evidence office gross weights for all the cards in a case do not total more than 28 grams, then only one item ( the largest one ) will be analyzed and no net weight will be determined. The remaining items will be not tested.

If the evidence office weights for all the cards in a case, total more than 28 grams, and the chemist believes the net weight of material may be greater than 28 grams, the chemist will analyze the largest item in the case and report a net weight. If the net weight of this item is greater than 28 grams, then the chemist does not need to analyze any other items\*. If the net weight of the largest item is less than 28 grams, the chemist will test and determine the net weight of enough items to insure a net weight of tested material greater than 28 grams.

\*In cases where several large vegetable matter items (example 5 items, one weighing 29 grams and four others each of 25 grams) are submitted, the chemist should notify a senior analyst or supervisor and they may decide to test more than one of the items.

MANY INSTANCES MAY NOT BE ADDRESSED HERE AND IN THOSE CASES, A SENIOR ANALYST OR SUPERVISOR SHOULD BE NOTIFIED TO DETERMINE A SAMPLING PLAN.

## TRAFFICKING CASES

For Marijuana trafficking items (greater than 50 pounds) a senior analyst or supervisor should be notified.

### 3) Pharmaceuticals

A) Class A and B controlled substances, excluding Oxycodone, Hydromorphone, Buprenorphine, Morphine and Codeine (class B).

All units (specimens) are similar ballistically, and presumptive identification can be determined by using the appropriate references, then for.....

1-50 units ----- 1 unit will be analyzed by GC-MS  
51-100 units----- 2 units will be analyzed by GC-MS  
101-200 units-----3 units will be analyzed by GC-MS  
If greater than 200 units notify senior staff chemist or lab supervisor

B) Oxycodone, Hydromorphone, Buprenorphine, Morphine and Codeine (class B) (opium derivatives)

All units (specimens) are similar by ballistics and presumptive identification can be made from the proper reference materials then:

- a) IF the net weight is less than 14.00 grams, then one unit will be tested by GC/MS.
- b) IF the net weight is = to or greater than 14 grams but less than 100.00 grams, then two (2) units will be analyzed by GC/MS.
- c) IF the net weight is= to or greater than 100.00 grams, then three (3) units will be analyzed by GC/MS.

C) Class C and Class D substances (all units/specimens are similar by ballistics) and identification can be made from appropriate reference sources, then for:

1-100 units.....analyze one unit by GC/MS  
101-200 units.....analyze 2 by GC-MS  
201-X units.....analyze 3 by GC-MS.

More testing may be done on any sample with the approval of the Lab Supervisor or a senior level analyst. If specimens (units) are in sealed containers, less sampling may be appropriate. The Laboratory Supervisor or a senior level chemist should be notified.

If a case contains several items of similarly marked Class C pharmaceuticals, then one item will be chemically tested and the others may be identified by Appearance and Labeling. The chemist will check with a supervisor before completing the case. The item with the most individual tablets will be chemically tested.

#### 4) Negatives

##### A) Substance/Powders

When testing an item that contains similar specimens and negative results occur, sampling will be done as follows:

1-10 specimens.....up to 5 will be tested

11 or greater.....2 X the square root of the total will be tested up to maximum of 20

When testing for barbiturates (Dilly-Koppanyi test), a composite of the specimens can be tested.

If a screening instrumental analysis is performed, then a composite of the specimens may be done.

##### B) Pharmaceuticals (negatives)

For pharmaceuticals that have similar markings, or for tablets or capsules that are similar in appearance, and that test negative, a minimum of three tablets or capsules should be screened. Color tests, a composite Dilly-Koppanyi, analysis, and a composite GC or UV Screening analysis, should be run.

##### C) Vegetable Material (negatives)

If vegetable material is similar by appearance and organoleptically, then the following sampling scheme will be used.

1-10 containers- 3 will be analyzed

11-x containers- 5 will be tested

For testing by Duquenois-Levine or GC testing composites may be done to rule out any positive material.

## Appendix C- Calculation of net weight

### Powders and Substances

- 1) Single specimen: the net weight will be determined, (page 6), and will be recorded on the chemists reporting sheet, and also on the lab control card. If the sample is weighed on a balance that measures to 3 or 4 decimal places, the chemist will truncate the net weight reading to 2 places and not round up. (for example, a resulting net weight of 1.2378 grams will be reported as 1.23 grams.
- 2) When reporting the net weight of a powder item, the minimum amount of powder that will be reported on a Certificate will be "less than .01 grams".
- 3) For items that have only one (unit) analyzed out of several, the net weight of the one unit tested will be reported as above. The Certificate will read the net weight of the analyzed item\* only  
\* Item here refers to an individual specimen or unit.
- 4) For samples where X units have been tested out of a total of N, an average for the net amount of substance in each unit will be determined. From that average, an estimated net weight for the entire exhibit will be extrapolated. The Certificate will read that N number of units was submitted, and that a representative number X, were tested. The total net weight for the entire exhibit (N units), was determined by an average of the net weight of the X sampled units. Example, a sample consisting of 22 similar bags is tested. According to the sampling plan in appendix B, 5 bags are chosen and net weights are performed on the five, with the following results:

Bag #	1 net weight	0.2154 grams
	2.	0.3516 g.
	3.	0.2908 g.
	4.	0.4720 g.
	5.	0.3507 g.
		-----
	total	1.6805 g divided by 5= 0.3361 g. ( average of one)
		22 x 0.3361g = 7.3942 grams

This will be truncated and reported as 7.39 grams, estimated net weight of 22 bags by 5 tested

\*\*\* NOTE that as of 9/12/2007 only 10% of the units need to be tested

- 5) The chemist should make every effort to calculate the items net weight by averaging the net weights of the individual units.
- 6) In cases where the amount of substance in each unit is quite variable, but the containers are uniform, the chemist may estimate the total net weight in the following manner. Example- 400 glassine bags have a gross weight of 138.34 grams. Twenty (20) specimens (bags) are tested. The weight of twenty empty bags is determined. The average weight of one empty container (bag) is calculated, and found to be 0.3252grams. Multiply the average empty container weight by 400 to get an estimated weight of the empty 400 bags.  $0.3252 \times 400 = 130.08$  grams. This is subtracted from the total gross weight of the 400 units (138.34 g), resulting in an estimated net weight of substance of 8.26 g.
- 7) Where various size units exist, for example small, medium, and large, then an estimated net weight for each size should be calculated. The reported net weight will be the sum of the estimated net weights of each size.

#### Appendix D Notarization of Certificates

Notarized Certificates of Analysis are prima facie evidence in the courts of Massachusetts. The chemist notarizing the certificate will record the certificate number, the person(s) whose certificates they are notarizing, and the time and date of the notarization. The notary will keep this information in a bound book with sequentially numbered pages.

#### Appendix E GC/MS Control Sheet (submittal for confirmatory testing)

- A) A separate control sheet will be prepared for each type of drug submitted except that different pharmaceuticals and steroids may be put on the same sheet
- B) Similar samples will be listed in numerical order
- C) Submitting agency will be identified
- D) A preliminary finding will be reported
- E) Any comments that may inform or help the GC-MS operator with their analysis will be reported in a comment section
- F) The Lab Control Card for each sample will be paper clipped to the GC-MS Control Sheet
- G) Each chemist will bring their sample vials to the GC-MS laboratory

#### Appendix F- References for Ballistic Identification of Pharmaceuticals

Physicians Desk References  
Micromedex Computer Search Program  
Department of Justice Logo Index  
Pharmacy REDBOOK  
State Laboratory Logo Compendium  
Drug Identification Bible

Other published references may be used with the supervisor's approval

## Appendix G Gas Chromatography (GC) Screen

### General Procedures

A) The sample will be dissolved in a proper solvent. This is usually Methanol for most substances. If in doubt the chemist should check the Merck Index, or Clarke's Identification of Drugs Manual, to determine the solubility characteristics of the suspected drug.

B) Blanks will be run before and after any standard that is run.

C) A blank will be run before each new sample vial. If the sample is composed of multiple vials, then a blank is not needed between each specimen number. In this instance, a blank should be run after each batch of 4-6 samples

D) A blank vial will be run as the last sample to be injected on the analysis run.

E) A Cocaine/Codeine standard should be run with unknowns to insure that Gas Chromatograph is operating correctly. The Retention Time (RT) of the sample should be compared to the RT's of the Cocaine/Codeine standard.

### 1) Unknown Powders, Substances, Tablets

Place 3-5 mg of sample into a standard GC vial. Fill the vial  $\frac{3}{4}$  full with Methanol and add 4-5 drops of Chloroform. Crimp seal and number vial. Run sample on a general-scan method using a HP-1, HP-5, or a similar column. Blanks and a Cocaine/Codeine standard should also be run. Note any retention times (RT) found in the sample, and compare to RT's for the standards. If any discernible GC peaks are seen for the sample, note relative RT to standards, and submit the sample to GC/MS laboratory for further testing\*.

If the GC/MS result comes back as positive for a controlled substance, then the primary chemist should review all previous analytical testing done on the sample, and check to see that preliminary results were indicative of the found substance. This can include review of, color tests, UV results or the preliminary GC result. If the color tests or UV are indicative of the drug, then the sample can be called positive. For the preliminary GC result, the chemist should note that the relative retention time of the controlled substance was similar in relation to the Cocaine/Codeine standard run with the preliminary GC. If a GC peak is found that corresponds to that expected of the substance, then this is a positive result and no further testing is necessary. If no corresponding GC peak was found in the original preliminary GC screen, then a new GC run should be performed using the standard that gave a positive result by the GC/MS.

- NOTE: Samples that elute quickly on the analytical columns used, such as Amphetamine or Methamphetamine, may not be seen on a general scan GC method. Suspicion of these types of drugs will be indicated by an orange to brown result with the Marquis test. A special Amphetamine GC method will be run to identify these drugs by Gas Chromatography.

## 2) Vegetable Matter (Marijuana)

Place 20-40 milligrams of vegetable matter into a clean tube. Add 1-3 mL of solvent to the tube and mix. Let the sample sit for five (5) minutes. Remove 1-2 mL of liquid and place in a standard GC vial. Run the sample along with blanks and Delta-9-THC standard on a HP-1, HP-5 or similar column using a ROUTINE, GEN SCAN or similar method. Note RT of sample and standards. If sample RT is within +/- 1.5 % to standard then test is positive by GC for Delta-9-THC. If the sample has a strong irritating odor, but does not test positive for THC, then it should be screened for PCP

## 3) Residues-

A residue volume GC vial (0.1- 0.3 mL) should be used. The sample will be placed in the residue vial, diluted with solvent, and run by an appropriate GC method, eg. Gen Scan, Routine, or Residue GC method on an appropriate column (see above). Blanks and a standard should be run. If GC peaks are present, vial should be sent to GC/MS for confirmatory testing along with a copy of the preliminary GC.

## Appendix H -Microcrystal tests

A Gold Chloride and TLTA microcrystalline test will be performed on all suspected Cocaine samples. These tests will be used to identify cocaine and differentiate the isomeric forms. Under Massachusetts law, only cocaine derived from the natural source is controlled. This is the L-isomer.

### 1) Gold Chloride-

Place a small amount of sample on a clean microscope slide-add one drop of dilute acetic acid. Add a drop of Gold Chloride crystal reagent to the side portion of the test solution. Allow mixture to stand for 30 - 60 seconds then examine for characteristic shaped X's. If no X's are observed, retest the sample. If no crystals are observed the second time or if crystals are distorted, proceed to GC testing of the sample to identify the presumptive presence of Cocaine or presence of interfering substances. See ASTM designation E 1968-98 Standard Guide for Microcrystal Testing in the Forensic Analysis of Cocaine (Addendum R)

### 2) TLTA test is used for determination of the L vs D isomer of Cocaine.

Once Cocaine presence is determined by the Gold Microcrystal test, the chemist will verify the presence of the L-isomer form of the cocaine. Place a small amount of sample on a glass microscope slide. Add a drop of TLTA reagent to the sample. Immediately observe the sample for characteristic sheaths of needles crystals. Sheaths of needles indicate the presence of L-Cocaine. If no needles are observed, the sample may be in the base form and not soluble. To dissolve sample, add a drop of dilute acetic acid to the mixture and gently mix. Observe crystals. Needles developing indicate the sample was in the base form. If no needle crystals are observed, perform a second crystal test. Place a small amount of sample on a glass microscope slide and add 1 drop of dilute HCl acid. Place the slide on a hot plate until the liquid evaporates. Remove slide and perform the TLTA test. Observe crystals. Sheaths of needles should develop. If no characteristic needles are observed at this time, a sample cleanup may be necessary and a senior analyst will be notified. See Microgram Vol. No 9, September 1982 (Addendum R)

\* For photographs of positive Gold and TLTA microcrystalline reactions see Addendum R.

## Appendix I- Color (spot) Tests

See Clarke's Isolation and Identification of Drugs & Poisons volumes 1-3, and the DEA Training Manual), for specific reagents.

Color tests are often done by placing a small amount of sample into a depression on a porcelain plate. A drop of reagent is then added and a color reaction change is noted. The chemist should note what reaction takes place in 3-5 seconds, as most color tests develop very rapidly. The chemist may also add reagent to the plate first and then add sample to the reagent.

Examples:

A) Cobalt Thiocyanate Test: Many substances give positive reaction to this test. A positive reaction is usually a blue color developing immediately after addition of reagent.

- 1) Cocaine HCl- strong flaky blue
- 2) Procaine and Lidocaine- pasty blue
- 3) Ketamine- weak blue
- 4) PCP powder weak blue
- 5) Heroin slight blue
- 6) Cocaine base (crack)- weak+, but strong blue with addition of dilute HCl acid

B) Marquis Reagent is a general test for several compounds  
Heroin, Morphine, Codeine---purple  
Amphetamine & Methamphetamine—orange to brown

C) Froehdes

D) Meckes

F) Dilly-Koppany- This test is performed by placing a small amount (5-10 mg) of sample into a test tube. Equal amounts (1-2 ml), of Dilly Koppany reagent and isopropyl amine are added. If derivatives of Barbatunic acid are present the liquid will turn purple.

Clarke's Isolation and Identification of Drugs has results for various color tests of most drugs.

## Appendix P Preliminary Testing Requests

### COCAINE and HEROIN

Preliminary Requests will come from the evidence office. They may be done on powder samples weighing greater than .01 grams. There must be sufficient sample so that preliminary results can definitely be verified by confirmatory testing. Residues will NOT be done on a preliminary basis. For preliminary results to be reported, the analyzing chemist must get positive preliminary test results as follows:

- Cocaine... a) Positive Cobalt/Acidified Cobalt Color test  
b) Positive Microcrystalline test  
c) Positive GC retention time match to Cocaine standard (+/- 1.5%)
- Heroin a) Positive Marquis, Froehdes, and Meckes color tests  
b) Positive GC retention time match to Heroin standard (+/-1.5%)

If multiple specimens are involved, chemists should check with a senior level chemist to determine if a preliminary net weight can be reported. If multiple specimens are involved, several specimens must be tested by color or microcrystal (for cocaine) tests, but only one specimen needs to be tested by GC analysis. All test results must be positive for the suspected substance.

If any tests are inconclusive, the chemist will notify the evidence office that the sample is not suitable for preliminary analysis, and further testing will be required.

#### Appendix T) Chemist's Initial Training Checklist

- 1) Chemist has been informed of the goals, mandates, and mission of the Department of Public Health Drug Analysis Laboratory.
- 2) Chemist has been made aware of the individual responsibilities of each individual chemist.
- 3) Chemist has been instructed in the procedure for submitting items to the laboratory.
- 4) Chemist has been instructed in the proper procedure for receiving samples from the Evidence Office.
- 5) Chemist has become familiar with the safety procedures followed in the laboratory. They have been instructed in the proper handling and dispensing of flammables and corrosives. They have been instructed in the use of a chemical fume hood and the operation of the laboratory autoclave.
- 6) Chemist has been instructed on the procedures to follow before opening and analyzing any item (sample).
- 7) Chemist has been instructed in the laboratory's sampling guidelines.
- 8) Chemist has been instructed in the use and care of laboratory balances.
- 9) Chemist has been instructed in the proper manner of calculating net weights.
- 10) Chemist has received training in performing color tests. The meaning of various results of these screening tests has been explained. The chemist has seen standards and samples run by the various reagents.
- 11) The chemist has been instructed in the use of a stereomicroscope and has examined the cystolith and glandular hairs of Marijuana.
- 12) The Chemist has been instructed in performing microcrystal tests. The use of a polarizing microscope has been demonstrated. The Cocaine microcrystal tests, the Aucl and the TLTA test, have been clearly explained. The chemist has seen standards and samples analyzed by this method.
- 13) The Chemist has been instructed in how to prepare samples for confirmatory testing (GC-MS). The use of proper sample size and the selection of the appropriate solvents has been clearly explained. The chemist has been instructed in the proper manner of preparing a GC-MS control sheet.
- 14) The Chemist has been instructed in the use of an Ultra Violet Spectrometer as a screening technique. The Chemist is able to prepare a powder sample and analyze it on this instrument.

15) The Chemist has been instructed on the use of a Gas Chromatograph. The chemist can prepare a powder sample for analysis, and can run the sample on the GC by the appropriate method. The proper use of standards and controls has been emphasized.

16) The Chemist has been instructed in the use of an Infra Red Spectrophotometer. The chemist can prepare a sample for this analysis. Emphasis on testing and determining Cocaine base from its salts was stressed.

17) The Chemist has become familiar with the Massachusetts statute dealing with controlled substances, MGL Chapter 94.

18) Storage and control of all analytical data, stressing storage by chronological order, and the use of standard forms has been explained.

19) All analytical tests that must be run, before a powder sample can be determined to be Negative, have been explained.

20) The QA/QC procedures followed in the Laboratory have been explained

21) All security procedures have been explained.

## U) Ultra-Violet Spectrophotometer Testing

### Unknown Powder samples.

Setup UV parameters to approximate the following:

Scan speed 100-240 wavelengths (/) / minute

Start / 350nm

End / 225nm

Absorbance 0-1.5

Peak pick= 3

Smoothing if available =15

Place about 1-2 mg of sample into a 16/100mm glass tube

Dilute with 2 mL of dilute Sulfuric Acid, approximately 0.1N

Run blank then Sample under same parameters

Note the maxima of any peaks and compare to known UV peak values listed in Clarke's Isolation of Drugs or other UV indexes

If the UV scan results in discernible peaks, further testing will be done. Example GC analysis (see Supervisor)

If no discernible peaks are identified, the test is negative

A UV standard such as an Ephedrine solution or a holmium oxide film should be run with each batch to insure proper functioning of instrument.

## Addendums

Addendum A .... SWGDRUG Code of Professional Conduct

Addendum B..... SWGDRUG Recommendation for Methods of Analysis/Drug Identification

Addendum L Chapter 94C MGL section 31-32

Addendum M Methods of Analysis

Addendum R References

Addendum A SWGDRUG Code of Professional Practice for Drug Analysts

Addendum B SWGDRUG Methods of Analysis/Drug Identification

Addendum L    Massachusetts General Law Chapter 94C sections 31 and 32

Addendum M Methods of Analysis for Other Category Items

## AMPHETAMINE and METHAMPHETAMINE

When preliminary color tests (orange to brown Marquis ) indicate the presence of an amphetamine type drug, then the sample will be screened by UV or GC testing., before submitting to GC/MS confirmation. A GC or a UV test will be run on each specimen being submitted to GC/MS, unless changed by the laboratory supervisor.

Determine Net Weight

Perform Marquis color test

### UV Analysis

- 1) Place 2-5 mg of sample in a test tube
- 2) Add 1-2 ml of dilute, 0.1N Sulfuric Acid to the tube
- 3) Run the sample by UV, scanning from 300-230 nm.,using normal operating conditions
- 4) Absorption peaks should match those of published references, + or – 2nm (see Clarkes) (251,257,263)
- 5)

### GC Analysis

The sample will be converted to the base form to achieve better GC results

- 2) Place 5-10 mg of sample into a 12 x 75 mm test tube
- 3) Add 1-2 ml of 0.1 N NaOH to the test tube. Cap with aluminum foil
- 4) Mix sample and then allow tube to stand for approx 2 hours. (conversion to base).
- 5) Add 1-2 ml of Hexane to the test tube. Cover tube with aluminum foil, and gently mix.
- 6) Allow layers to separate. Hexane is on the top.
- 7) Carefully remove the top layer (hexane) from the tube with a Pasteur pipet, and place in a standard GC vial.
- 8) The sample will be analyzed by GC, using the ROUTINE GC method. A cocaine/codeine standard or an amphetamine or methamphetamine standard will be run with the samples.
- 9) Note retention time of sample to standards.
- 10) If concentration of compound of interest approaches that of the standards, the sample can be submitted to GC/MS as is. If sample is too strong or weak in comparison to the standards, then chemist will adjust to appropriate level.
- 11) If RT is comparable to Amphetamine or Methamphetamine standard, then the sample will be submitted to GC/MS as either a suspected Amphetamine or Methamphetamine drug. If only a cocaine /codeine standard is run, with the sample then the chemist should note on their GC/MS control sheet that the suspected drug is an early eluter.

COCAINE: Powder / Substance

1) Perform evidentiary check to verify item identification

2) Determine evidence gross weight

3) Determine sampling plan

If a statistical inference is to be made of the entire population of the item, then the Hypergeometric sampling plan will be used. If a statistical estimate of the entire population is not needed, the chemist will test 10% or the square root of the entire population.

4) Determine Net Weight

5) Perform the Cobalt Thiocyanate, Marquis, Froedhes ,and Meckes color tests

6) Perform Aucl Microcrystal test

7) Perform TLTA Microcrystal test

8) If preliminary tests are positive for Cocaine, submit an aliquot for Gas Chromatography/Mass Spectrum analysis

If the Cobalt Thiocyanate color test, Aucl and TLTA microcrystal tests, and GC/MS tests are all positive, the sample is identified as Cocaine

HEROIN Powder/ Substance

1) Perform evidentiary check to verify item identification

2) Determine evidence gross weight

3) Determine sampling plan

If a statistical inference is to be made of the entire population of the item, then the Hypergeometric sampling plan will be used. If a statistical estimate of the entire population is not needed, then the chemist will test 10% or the square root of the entire population.

4) Determine Net Weight

5) Perform the Cobalt Thiocyanate, Marquis, Froedhes and Meckes.color tests.

7) If preliminary color tests are positive for heroin, submit an aliquot for GC/MS confirmatory testing

If the Marquis, Froedhes and Meckes color tests, and the GC/MS are positive for Heroin, the sample is identified as Heroin

## KETAMINE:

### A) In liquid form

If suspected Ketamine is in liquid form and is labeled Ketamine HCl, (such as in an injectable), then an aliquot will be evaporated and the residue will be analyzed by GC/MS. If the sample is positive by GC/MS for Ketamine, then the sample will be determined to contain Ketamine HCl.

If a liquid is suspected as being Ketamine and is not labeled as the HCl, an aliquot will be evaporated and the residue will be analyzed by GC/MS. If the sample tests positive for Ketamine by GC/MS, then it will be further tested by IR analysis to determine the salt form.

### B) In powder form

Suspected Ketamine powder samples will be handled as all routine powders are handled. If preliminary testing indicates the possible presence of Ketamine, then a GC or UV analysis will be performed. If positive by either testing method, see Clarke's for GC and UV values, then an aliquot of sample will be submitted for GC/MS confirmation. If positive for Ketamine by GC/MS, then a portion of the sample will be analyzed by IR to determine the salt form.

## MDMA (3,4-Methylenedioxymethamphetamine) or MDA (3,4-Methylenedioxyamphetamine)

### In Tablet Form

After verification of sample as in Appendix A, the sample will be opened and a description of the sample including: color, size, shape, count, and markings will be recorded. A drawing of the tablet will also be included. The information will be recorded on a powder sheet or in a bound book.

Number of specimens tested will be determined by following the sampling plan for Pharmaceuticals (class A and B), non-opium derivatives.

- 1) A portion of a tablet or capsule will be ground to powder (use 1/4 to 1/2 of a tablet)
- 2) Color tests will be performed on the powder. The tests should include Cobalt Thiocyanate, Marquis, Froehdes, and Meckes tests. Typical positive reactions for MDMA or MDA are:

Marquis- purple to black, Froehdes- green to black, Meckes-green to dark bluegreen

(see Clarke's Isolation and Identification of Drugs, volumes 1-3).

- 3) If tests are questionable, the sample will be handled as an unknown powder sample.

If color tests are positive, place 5-10 mg of the sample into a 12 x75 mm test tube. Add 1-2 ml of methanol and vortex for 10-20 seconds. Let stand for 20-30 minutes,

- 4) Decant the liquid into a standard GC vial and cap..

- 5) A GC screen will be run with a MDMA, a MDA, and a Cocaine/Codeine standard.

6) If the retention time (RT) from the GC test indicates the presence of MDMA or MDA, submit the GC vial to GC-MS for confirmatory testing in the normal manner. If the GC retention times indicate a substance other than MDMA or MDA, submit the sample to GC/MS as an unknown.

PHENCYCLIDINE (PCP): Submitted as Vegetable Matter

- 1 Item will be verified as in Appendix A
- 2 A gross weight of the item will be done and observations will be recorded on a powder sheet or in a bound book.
- 3 Sampling procedure is the same as a non trafficking powder sample.
- 4 Organoleptic observation will be done. PCP usually has a strong pungent odor.
- 6) A small amount of vegetable material (10-20 mgs.) will be placed in a disposable test tube (approximately 12mm x 75mm.).
- 7) 1.0-2.0 ml of methanol will be added to the tube.
- 8) Cap the tube with aluminum foil, and then vortex for 10-20 seconds.
- 9) Place the tube in a rack and let stand for 20-30 minutes.
- 10) After standing, pipet the methanol into a standard GC vial and cap.
- 11) A preliminary GC will be run using a routine method. A cocaine/codeine standard and a PCP standard should be run with the sample.
- 12) If a GC Retention time (RT) of the sample chromatogram matches the RT of the PCP standard, then submit the sample vial to GC/MS for confirmation of the presence of PCP. If the sample chromatogram has a peak that does not match the PCP standard, then submit to GC/MS as an unknown. If no GC peaks are detected, the sample can be called Negative.



General procedures for analyzing routine unknown Substances / Powders

- 1) Determine Gross Weight of item
- 2) Determine if sampling plan is appropriate
- 3) Determine Net Weight
- 4) Perform preliminary color testing of substance.

If sample amount allows, chemist should do the following preliminary color tests

- a) Marquis
- b) Froehdes
- c) Meckes
- d) Cobalt Thiocyanate
- e) Dilly-Koppanyi

If preliminary color tests indicate the possible presence of Cocaine, then the chemist will perform separate Microcrystal tests using Gold Chloride, and TLTA reagents

If microcrystal testing indicates cocaine, then a portion of the sample will be submitted to Gas Chromatography/Mass Spectroscopy (GC/MS) for confirmation.

If color tests indicate the presence of another controlled substance, or if the microcrystal tests are inconclusive for Cocaine, then the chemist will do further instrumental testing, or submit a portion of the sample to GC/MS, for confirmation

All unknown powder/substance items will be confirmed by GC/MS.

## Alcohol (Ethanol) Determination

### Qualitative

- i. check that number on submission sheet matches number on sample container
- ii. Note approximate amount of liquid in sample
- iii. Note odor of liquid by carefully wharfing hand over opened bottle toward nose
- iv. Using a Pasteur pipette, transfer 1-2 ml of sample to a standard GC vial (note color and appearance of sample).
- v. Using a 10 ul syringe inject approximately 0.2 ul of sample into Gas Chromatograph\*.
- vi. Compare retention time of sample to a similarly injected 10%, and an a 30% ethanol standard. Percentage are approximate.
- vii. If retention time of sample and standard are similar proceed to quantitation section.\*\*

#### \* GC parameters

- 1) Column- 6 ft packed porapak# Flow 40-50 ml/min helium 165-180 C.
  - 2) Injector- 185-195 C
  - 3) Detector- 200-220 C
- or

\* use appropriate capillary column with current alcohol method

Blanks of water will be injected between different samples and standards. The syringe will be thoroughly rinsed with water after each injection.

#### \*\* Quantitation:

Note the relative GC response of the sample to the 10% and 30% standards. If the sample GC response is similar to the 10% standard, then a 2X dilution is appropriate. If the GC response of the sample is similar to the 30 % standard, then a 10X dilution is appropriate. Samples will be diluted with deionized or distilled water. The chemist will add 5ml of sample to either a 25 or 50 ml volumetric flask, depending on the estimated sample concentration. Tthe chemist will then pipet 1 or 2 ml of Isopropanol (internal standard) into a clean 25 ml or 50 ml volumetric flask. The flask will then be brought to volume with water. A standard GC vial will be filled with this solution. The sample will be run in triplicate by GC under the same conditions as in the qualitative analysis

See AOAC Official Methods of Analysis 1990 for reference of this method

Following quantitation, the diluted samples containing ISTD will be sent to GC/MS for the confirmation of the presence of ethanol

## NITRITE ANALYSIS

Liquids suspected of containing a controlled nitrite, (see Massachusetts Controlled Substance Law Chapter 94 C), will be tested by a color test, a screening instrumental test, and a confirmatory GC/MS analysis.

### Equipment

- A) Ultraviolet Spectrophotometer (UV)
- B) Gas Chromatograph (GC)
- C) GC/Mass Spectrophotometer (GC/MS)
- D) Chemical Fume Hoods

### Glassware

- A) glass test tubes (16 x 100 mm)
- B) 2 mL glass GC sample vials
- C) 9 inch Pasteur pipets

### Chemicals:

- Methanol (reagent grade)
- Methylene Chloride (HPLC grade)
- Amyl or Iso-Amyl Nitrite stock solution
- Butyl or N-Butyl Nitrite stock solution
- Iso-Butyl Nitrite stock solution
- Cyclohexanol
- 95 % Ethanol
- Isopropyl Nitrite stock solution

### Reagents:

Nitrite test strips (Mercoquant) or similar No2 test strips

## STANDARDS

A) GC standards- standards should be prepared fresh with each batch of samples to be run. The corresponding alcohol will be prepared for GC testing. See attachment 1

- viii. Amyl Nitrite- using a Pasteur pipet place 2 drops of Amyl or Iso-Amyl Nitrite stock solution into a GC vial. Bring to volume with methanol and crimp seal. Let stand for 10 minutes before using.
- ix. Butyl and Iso-butyl standard will be prepared in the same manner as above, using the appropriate stock solution.
- x. Cyclohexanol standard is prepared by adding 3 drops of cyclohexanol to a GC vial and bringing to volume with methanol. This standard last indefinitely and does not have to be prepared fresh.
- xi. Isopropyl nitrite does not have to be run with each batch of samples.

B) UV Standards – A single UV standard will be prepared fresh for each batch of samples analyzed

Place two drops of Iso-butyl nitrite standard into a glass tube. Add 2 mL of methanol, cover with aluminum foil, and let stand for 10 minutes before using.

C) GC/MS standards

Standards are prepared by pipeting 2 drops of the proper nitrite stock solution into a GC vial. Bring the vial to volume with Methylene Chloride. An Amyl or Iso-Amyl nitrite , as

well as a Butyl and iso-butyl nitrite standards will be prepared with each batch of samples to be analyzed.

Analysis:

1) Organoleptic test: The liquid in the sample bottle will be warfed toward the chemist to see if any strong odor is detected.

2) A Nitrite color strip test will be performed on each sample. A new strip will be used for each sample. The raised lower portion of the strip will be immersed into the sample for one second and then be removed. The raised area of the strip will quickly turn purple if NO<sub>2</sub> are present. Follow the manufacturers instructions.

3) If NO<sub>2</sub> test is positive place 3 drops of sample into test tube, and add approximately 2 ml of methanol. Cap with aluminum foil and let stand for 5-10 minutes. If Nitrite is present it will be converted to the corresponding alcohol. After sitting, run the liquid on a scanning UV Spectrophotometer, using the same parameters as those run for the various NO<sub>2</sub> standards. Compare spectrum to standard. A distinctive 5 fingered spectrum should result if the corresponding alcohol is present. Several dilutions of the converted alcohol may be needed before a meaningful spectrum is obtained.

## Pharmaceuticals

In general the goal in pharmaceutical analysis is arrive at a concentration of analyte of approximately 1 mg/ml for confirmation by GC/MS.

### Cleanup Methods

Acid shakeout: Place 15-20 ml of deionized water into a standard separatory funnel. Add appropriate amount of sample to be cleaned up. Add 3-5 drops of concentrated Hydrochloric Acid. Add 15-20 ml of chloroform. Gently invert the funnel several times to mix. Remove chloroform (bottom) layer into a beaker.

Base Shakeout: Place 15-20 ml of deionized water into a standard separatory funnel. Add approximately 100mg of bicarbonate to the water, and mix. Add an appropriate amount of sample to the solution and then add 15-20 ml of chloroform. Gently invert funnel to mix. Remove the bottom layer (chloroform) into a beaker.

Extraction methods using other solvents is also acceptable

### Typical Pharmaceuticals

(Butalbital)

The cleanup for Barbitol when mixed with Caffeine and Acetaminophen, calls for an Acid shakeout. Gently evaporate the chloroform. Depending on the starting concentration of Barbitol, bring up the residue in the beaker with Methanol and place into a standard GC vial.

(Buprenorphine) Suboxone

Cut 1- 8mg suboxone tablet in half. Crush and place in disposable test tube and add 3-4 ml of methanol. Allow to settle for several hours. Remove methanol into a standard GC vial

(Clonazepam)- .5-1.0 mg tablet

Crush 1 tablet, and place it into a 12 x 75 mm disposable test tube. Add approximately .5 to 1ml of acetone and mix. Allow to settle. Decant of approximately .1ml into a Residue GC vial.

(Codeine, Hydrocodone, Oxycodone /Acetaminophen)

Crush up a tablet and perform a Base shakeout. Evaporate the chloroform. Add an appropriate amount of methanol to the beaker, depending on the concentration of codeine in the tablet.

Addendum R

REFERENCE MATERIALS

Clarke's Isolation and Identification of Drugs Volumes I, II, and III  
Journal of Forensic Science  
Journal of Chromatography  
DEA Training Manual 1982  
Bulletins of the ASTM International  
Recommendations of the Scientific Working Group for Drug Identification  
AOAC Official Methods of Analysis vol 13  
Internal Revenue Service Methods of Analysis

Training Guideline Manual

First Draft compiled 9/01/2005  
Second Draft compiled 9/2/2008

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## Training Guidelines for New Chemists

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